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Indian Standard

METHODS OF DETERMINATION OF MASS OF TIN COATING ON TINPLATE

(Third Revision)

Hot Dip, Sprayed and Diffusion Coatings Sectional Committee, SMDC 28 [Ref : Doc : SMDC 28 (3072)]

1. Scope

1.1 This standard prescribes three test procedures for determination of mass of tin coating on hot and electrolytic tinplate as follows:

a) Weight loss method,

b) Bendix method, and

c) Referee method.

1.2 The supplier or the purchaser may, however, use any of the three methods described in this standard or any other suitable instrumental method for determining the mass of tin coating on tinplates for the purpose of control at their respective ends. However, in case of dispute, the referee method shall be used for determination of mass of tin coatings.

2. Sampling

2.1 For the purpose of determination of mass of tin coating on tinplate, samples shall be drawn in accordance with the procedure described in IS : 1993-1982 'Specification for cold-reduced tinplate and cold-reduced blackplate (*second revision*)'.

2.2 *Preparation of Samples* — The test specimen or areas to be sampled should be free of visible contamination by oil, grease or other substance that would interfere with dissolving of tin from the sample surface.

2.2.1 The samples may be cleaned by cathodically treating in 0·5 percent sodium carbonate solution for 10 seconds at a current density of 0·077 5 amperes/cm^2.

2.2.2 Alternatively, the samples may be cleaned by wiping the surface of the sample with a clean cloth dipped in reagent grade acetone followed by immersion of sample in acetone and finally through drying.

2.2.3 Any convenient size of the test specimen may be taken but it should be a minimum of 50 cm^2 for weight loss method and 25 cm^2 for Bendix and referee method.

3. Weight Loss Method

3.1 *Outline of the Method* — The tin coating on a previously weighed sample of tinplate of known area is selectively removed from steel base by hydrochloric acid inhibited with antimony trichloride. The difference in weight after the removal of tin coating is determined and expressed in terms of unit area.

3.2 *Reagents*

3.2.1 All reagents shall be of the highest purity obtainable and distilled water (*see* IS : 1070-1977 'Specification for water for general laboratory use (*second revision*)' should be used throughout. Solution shall be freshly prepared and, where necessary, filtered.

3.2.2 *De-tinning solution* — Dissolve 20 g of antimony trioxide in 1 litre concentrated hydrochloric acid (r d = 1·16).

3.3 *Procedure*

3.3.1 Take cleaned specimen (*see* **2.2**) of size 50 × 50 mm (in case of electrolytic tinplate of coating E 5·6/5·6 or below) or 5 × 10 cm (in case of hot dipped tinplates and electrolytic tinplate of coating above E 5·6/5·6). Rinse thoroughly followed by complete drying. Weigh specimen accurately (m_1).

Adopted 20 February 1988

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3.3.2 Dip the specimen in de-tinning solution and keep it immersed till about 1 minute after all gas evolution has stopped.

3.3.3 Remove specimen from solution and wash immediately in running water. Remove completely the loosely adherent deposit of antimony by mopping with a soft cotton wool swab.

3.3.4 Rinse thoroughly and completely dry the specimen before weighing (m_2).

3.4 Expression of Results — The mass of tin coating on tinplate is obtained from the following formula:

$$\text{Mass of tin coating in g/m}^2 = \frac{(m_1 - m_2) \times 10^6}{A} - C$$

m_1 = mass of specimen (in grams) prior to de-tinning,

m_2 = mass of specimen (in grams) after de-tinning,

A = area of specimen in mm², and

C = correction factor for iron dissolved along with tin from iron tin alloy layer.

The value of C is given by:

C = 0·4 g/m² (for flow melted electrolytic tinplates),

C = 1·4 g/m² (for hot dipped tinplate), and

C = 0 (for unmelted electrolytic tinplate).

3.5 Procedure for Differentially Coated Tinplate — For determining the tincoating mass of individual face of differentially coated tinplate, one face of the sample disc is blocked by cellulose lacquer, keeping the other surface exposed to de-tinning solution. The tincoating mass of the exposed surface is then determined in accordance with **3.3** and **3.4**, taking correction face factor C as 0·20. The tin coating mass of the blocked surface is then determined after removing the lacquer by a suitable solvent.

4. Bendix Method

4.1 *Outline of the Method* — The procedure involves dissolution of tin from a tinplate anode in dilute hydrochloric acid solution containing a measured excess of standard potassium iodate potassium iodide solution Excess iodine from the iodate-iodide solution is back titrated with standard sodium thiosulphate solution using a starch indicator.

4.2 *Reagents*

4.2.1 All reagents shall be of the highest purity obtainable and distilled water (*see* IS : 1070-1977) should be used throughout. Solutions shall be freshly prepared and, where necessary, filtered.

4.2.2 *Dilute Hydrochloric acid solution* (*1·7* to *2·0 N*) — Add one part of concentrated hydrochloric acid (r d = 1·16) conforming to IS : 265-1976 'Specification for hydrochloric acid (*second revision*), to 5 parts of water and mix well.

4.2.3 *Concentrated hydrochloric acid solution* (*1:1*) — Add one part of concentrated hydrochloric acid (r d = 1·16 conforming to IS : 265-1976) to one part of water and mix well.

4.2.4 *Aluminium metal* — 99·99 percent purity (tin free), as foil, 0·25 mm thickness.

4.2.5 *Ferric chloride solution* (*100 g/l*) — Dissolve 100 g of hydrated ferric chloride in water containing 100 ml of concentrated hydrochloric acid (r d = 1·16) and dilute 1 000 ml with water.

4.2.6 *Tin metal* — 99·95 percent or purer.

4.2.7 *Carbon dioxide gas* — Oxygen free.

4.2.8 *Starch solution* — Make a suspension of 1 g of soluble starch in 10 ml of water and add to 100 ml of boiling water. Boil for 2 or 3 minutes and cool.

4.2.9 *Standard potassium iodate-iodide solution* (*approx 0·0975 N*) — Dissolve 3·4 g of potassium iodate, 21·74 g of potassium iodide and 1·21 g of sodium hydroxide in 1 litre of distilled water. To standardize the solution, transfer an accurately weighed quantity of tin to a 500-ml Erlenmeyer flask. Add 200 ml of concentrated hydrochloric acid solution. Connect the flask to a carbon dioxide system and pass carbon dioxide for 5 minutes to displace air in the flask. While continuing the flow of carbon dioxide, heat the flask but do not boil violently. After the tin has dissolved, add 10 ml of ferric chloride solution and 2 g of aluminium metal foil. Heat carefully to boiling until the aluminium is completely dissolved and continue for additional 5 to 10 minutes after dissolution. Cool the flask to room temperature in running water while maintaining an atmosphere of carbon dioxide. Disconnect from the carbon dioxide system and titrate with potassium iodate-iodide solution using 5 ml starch solution as an indicator. Calculate the tin titre, T_1, as follows:

$$T_1 = A/B$$

where

A = grams of tin used,

B = millilitres of potassium iodate-iodide solution used for titration, and

T_1 = tin titre (grams of tin/ml) for potassium iodate-iodide solution.

4.2.10 *Standard sodium thiosulphate solution (for coatings above E 2·8/2·8)* — Dissolve 15·11 g of sodium thiosulphate ($Na_2S_2O_3.5H_2O$) and 1·11 g of sodium hydroxide in 1 litre of distilled water in a light proof bottle. Allow solution to age for 72 hours and remix. To standardize the solution, titrate against 20 ml potassium iodate-iodide standard solution and note the titre value T_2 (in ml).

4.2.11 *Standard sodium thiosulphate solution (for coatings E 2·8/2·8 and under)* — Dissolve 6·57 g of sodium thiosulphate ($Na_2S_2O_3.5H_2O$) and 2·78 g of sodium hydroxide in 1 litre of distilled water in a light proof bottle. Allow solution to age for 72 hours and remix. Standardize by the same procedure used for the stronger sodium thiosulphate solution but use only 10 ml of potassium iodate-iodide standard solution and note the titre value T_3 (in ml).

4.3 Apparatus

4.3.1 The de-tinning cell consists essentially of two cathodes of carbon rod, the sample that serves as an anode, and a beaker of dilute hydrochloric acid electrolyte. The carbon rods (152 × 6·35 mm) encased in porous porcelain thimbles are suspended from a suitable frame about 25 mm apart. A small glass or teflon enclosed magnet is attached to the frame in such a manner that it will hold the sample suspended midway between the two cathodes. A movable platform permits the beaker of electrolyte to be brought up around the assembly so that the sample may be completely immersed (*see* Fig. 1)

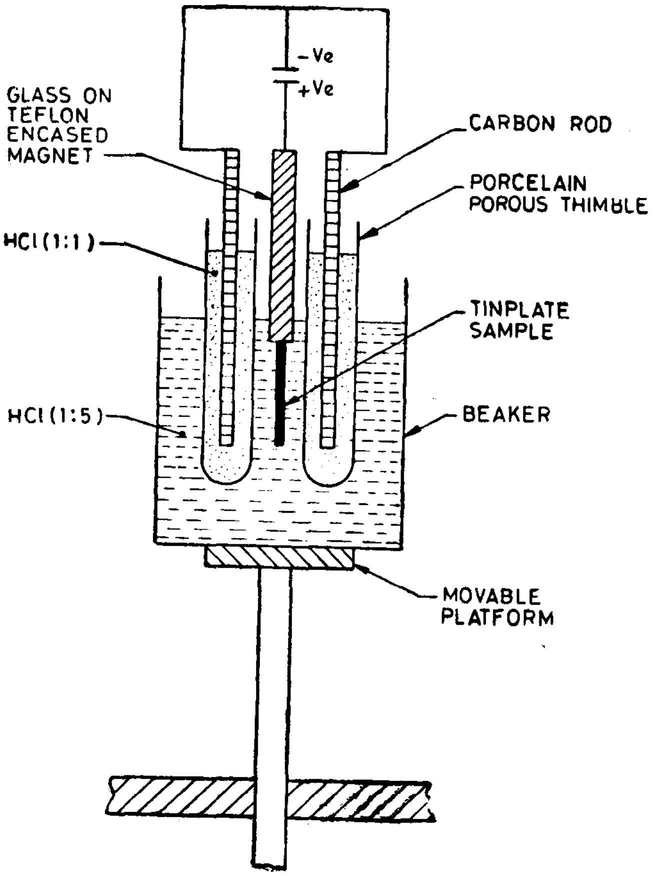

FIG. 1 DETINING CELL FOR BENDIX METHOD

4.3.2 A source of direct current that can be regulated to supply up to 3 A at 3 to 5 V through the deplater is required.

4.3.3 Although regular laboratory glassware may be used, it is advisable to use automatic dispensing pipettes or burettes, a motor-driven stirrer for titration, and a timing switch when a large number of determinations to be made.

4.3.4 *Precautions* — The apparatus must be kept in continuous operation to prevent iron in the solution adhering to the porous cells from oxidizing and subsequently liberating iodine from the potassium iodate-potassium iodide solution. If the instrument has been idle for some time, it is necessary to remove the ferric iron by running a disc of tinplate through the regular procedure before test samples are run.

4.4 *Procedure*

4.4.1 Make the required electrical connections.

4.4.2 Add concentrated hydrochloric acid solution to the porous tubes containing the carbon cathodes.

4.4.3 Suspend the cleaned sample (*see* **2.2**) of tinplate from the magnetic holder.

Note — If it is desired to strip on one side only (as for differentially coated electrolytic tinplate), mask the opposite side and reduce the current to half its normal value.

4.4.4 Take a measured quantity of standardized potassium iodate-iodide solution into a 400-ml beaker. The quantity of solution to be taken depends on the coating weight (*see* **4.4.8**). Add 250 ml of dilute hydrochloric acid solution and mix thoroughly.

4.4.5 Raise the beaker so that the sample and the porous cells are immersed.

4.4.6 Switch on the current and adjust to give a current density of 0·077 5 amperes/cm².

4.4.7 Switch off current after all the tin is removed. The stripping time is dependent on coating mass (*see* **4.4.8**). Stripping time should not be longer than is required to remove all the tin. Results will be higher by approximately 0·22 g/m² for each minute of overstripping.

4.4.8 The amount of KIO_3 solution (*see* **4.4.4**) and stripping time (*see* **4.4.7**) to be used are as follow:

Product	*Stripping Time*	*Amount of Standard Potassium iodate Solution*
		ml
Hot dipped tinplate H 14/14	90	20
Electrolyte tinplate E 11·2/11·2	90	20
Electrolyte tinplate E 8·4/8·4	75	20
Electrolyte tinplate E 5·6/5·6	60	10
Electrolyte tinplate E 2·8/2·8	60	10

4.4.9 Remove the beaker and add approximately 5 ml of starch indicator solution.

4.4.10 Titrate with standardized sodium thiosulphate solution to the disappearance of the blue colour. Note the titre value T_4 (in ml).

4.5 *Expression of Results* — The mass of tin coating on tinplate is obtained from the following formula:—

$$\text{Mass of tin coating in g/m}^2 = \frac{T_1\,(\,T_2 \text{ or } T_3 - T_4\,) \times 10^6}{A}$$

where

T_1 = tin titre value in g/ml of standard potassium iodate-iodide solution,

T_2 or T_3 = titre value in ml of sodium thiosulphate required for 20/10 ml of standard potassium iodate-iodide solution, and

A = area of sample (in mm^2).

5. Referee Method

5.1 *Outline of the Method*

5.1.1 The tin coating is dissolved in hydrochloric acid and the tin is reduced to bivalent state with metallic aluminium. The tin in the reduced state is determined by titration with standard potassium iodate-iodide solution.

5.1.2 The effective range of the method is from 2·5 g/m^2 up to 50 g/m^2 and the reproducibility is ± 0·1 g/m^2.

5.2 *Reagents*

5.2.1 All the reagents shall be of highest purity obtainable and distilled water (*see* IS : 1070-1977) should be used throughout. Solutions shall be freshly prepared and where necessary, filtered.

5.2.2 *Dilute hydrochloric acid (750 ml/l)* — Dilute 750 ml of concentrated hydrochloric acid (r d = 1·16) to 1 000 ml with water.

5.2.3 *Concentrated hydrochloric acid* (r d = 1·16) conforming to IS : 265-1976.

5.2.4 *Ferric chloride solution (100 g/l)* — Dissolve 100 g of hydrated ferric chloride in water containing 100 ml of concentrated hydrochloric acid (r d = 1·16) and dilute to 1 000 ml with water.

5.2.5 *Platinum wire* — A length of approximately 750 mm of 0·6 mm diameter platinum wire is formed into a flat spiral of two turns and approximately 125 mm diameter (*see* Fig. 2).

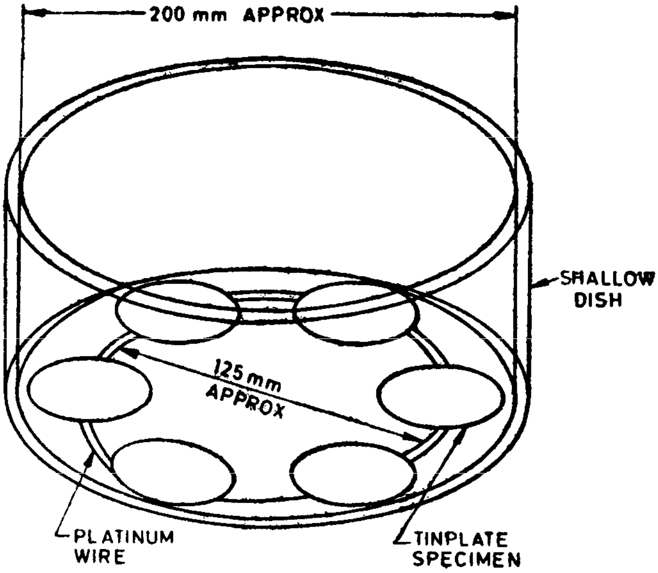

FIG. 2 ARRANGEMENT OF SPECIMENS FOR DISSOLUTION OF TIN

5.2.6 *Aluminium metal* — 99·99 percent purity (tin free), as foil of 0·25 mm thickness.

5.2.7 *Carbon dioxide gas* — Oxygen free.

5.2.8 *Lacquer* — A suitable air drying cellulose lacquer.

5.2.9 *Starch solution* — Make a suspension of 1 g of soluble starch in 10 ml of water and add to 100 ml of boiling water. Boil for 2 or 3 minutes and cool.

5.2.10 *Tin metal* — 99·95 percent or purer.

5.2.11 *Standard potassium iodate-iodide solution approx 0·05 N (solution 1) for use with electrolytic tinplate equally coated* — Dissolve 1·783 5 g of potassium iodate (previously dried to constant mass at 180°C) and 10 g of potassium iodide in water containing 0·5 g of sodium hydroxide and dilute to 1 000 ml with water.

5.2.12 *Standard potassium iodate-iodide solution approx 0·025 N (solution 2) for use with electrolytic tinplate differentially coated* — Dissolve 0.891 8 g of potassium iodate (previously dried to constant mass at 180°C) and 10 g of potassium iodide in water containing 0·5 g of sodium hydroxide and dilute to 1 000 ml with water.

5.2.13 *Standard potassium iodate-iodide solution 0·10 N (solution 3) for use with hot-dipped tinplate* — Dissolve 3·567 0 g of potassium iodate (previously dried to constant weight at 180°C) and 37 5 g of potassium iodide in water containing 0·5 g of sodium hydroxide and dilute to 1 000 ml with water (1 ml of this solution = 0·005 935 g Sn).

5.2.14 *Standardization of potassium iodate-iodide solution* — Transfer an accurately weighed quantity of tin (approximately 0·1 g) to a 500 ml Erlenmeyer flask. Add 100 ml of concentrated hydrochloric acid. Connect the flask to a carbon dioxide system and pass carbon dioxide for 5 minutes to displace the air in the flask. While continuing the flow of carbon dioxide, heat the flask but do not boil violently. After the tin has dissolved, add 10 ml of ferric chloride solution and 2 g of aluminium metal foil. Heat carefully to boiling until the aluminium is completely dissolved and continue for an additional 5 to 10 minutes after dissolution. Cool the flask to room temperature in running water while maintaining an atmosphere of carbon dioxide. Disconnect from the carbon dioxide system and titrate with the appropriate potassium iodate-iodide solution using 5 ml starch solution as an indicator. Calculate the tin titre, *T*, as follows:

$$T = A/B$$

where

A = grams of tin used,

B = millilitres of potassium iodate-iodide solution used for titration, and

T = tin titre value (grams of tin/ml) for potassium iodate-iodide solution used.

5.3 *Apparatus*

5.3.1 A suitable assembly for carrying out reduction of tin consists of a 500-ml wide neck conical flask marked at a volume of 200 ml. The flask is fitted with a rubber bung containing a bent gas inlet tube, a small Liebig type condenser and a rubber sealed tube for burette entry at the titration stage (*see* Fig. 3).

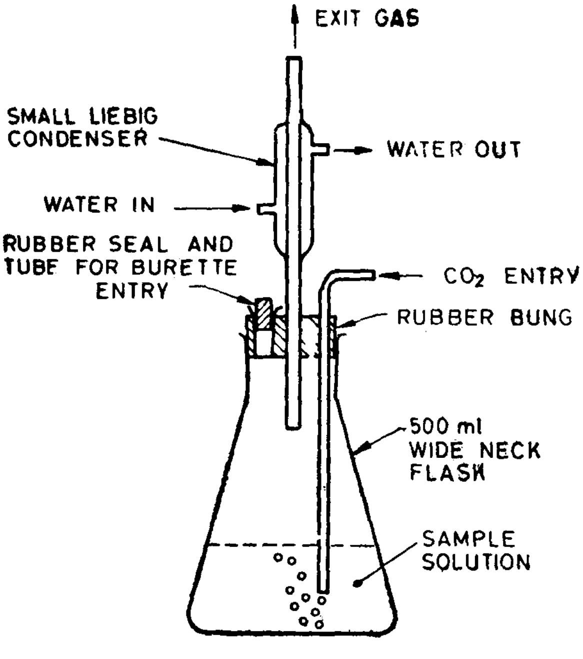

FIG. 3 APPARATUS FOR THE REDUCTION OF TIN

5.4 *Procedure*

5.4.1 *For electrolytic tinplate equally coated*

5.4.1.1 Take the clean specimen (*see* **2.2**) from four sheets sampled as in Table 7 of IS : 1993-1982: Place the spiral of platinum wire centrally in a shallow dish (*see* Fig. 2). Place six of the discs in a circle on the platinum wire and carefully pour 150 ml of dilute hydrochloric acid into the dish. As soon as the coating is completely dissolved from both the faces, leaving the steel surfaces exposed (*see* note under **5.4.1.3**), transfer the acid quantitatively to a 1 000 ml volumetric flask. Wash twice with 25 ml of water transferring the washings to the flask. Repeat the whole procedure with the remaining six discs, combining the acid and washings in the same volumetric flask, finally diluting to the mark with water.

5.4.1.2 Transfer a 100-ml aliquot of the solution to the 500-ml wide neck conical flask, add 75 ml of concentrated hydrochloric acid and 10 ml of ferric chloride solution and dilute to the 200-ml mark with water. Add 2 g of aluminium metal foil. Insert the rubber bung fitted with a small Liebig condenser, a carbon dioxide entry tube and a rubber sealed burette entry tube (*see* Fig. 3). Connect the apparatus to the appropriate supply points and pass carbon dioxide gas for 5 minutes to displace the air within the flask. Heat carefully to boiling avoiding vigorous evolution of hydrogen. Continue boiling for 5 to 10 minutes after dissolution of the aluminium metal. Cool quickly to less than 20°C maintaining an adequate supply of carbon dioxide.

5.4.1.3 Remove the burette entry tube seal, add 5 ml starch solution and titrate with standard potassium iodate-iodide solution 1 to a permanent blue colour.

Note — The time required for complete dissolution depends on the mass of coating. It may vary from about 3 minutes to about 15 minutes.

5.4.2 *For electrolytic tinplate differentially coated*

5.4.2.1 Take the clean specimen (*see* **2.2**) from four sheets sampled as in Table 7 of IS : 1993-1982 and coat the faces, carrying the tin coating with the greater mass, with a suitable cellulose lacquer. Allow to dry for 15 minutes, apply a second cost of lacquer and allow to dry for 1 hour. Place the spiral of platinum wire centrally in a shallow dish (*see* Fig. 2). Place six of the discs in a circle with the unlacquered faces in contact with the platinum wire. Carefully pour 150 ml of dilute hydrochloric acid into the dish. As soon as the coating is completely dissolved from the unlacquered faces, leaving the steel surfaces exposed, transfer the acid quantitatively to a 1 000-ml volumetric flask. Wash twice with 25 ml of water, transferring the washings to the flask. Repeat this whole procedure with remaining six discs. Combining the acid and washings in the same volumetric flask, finally diluting to the mark with water. Dry the discs and keep them for determination of the coating on the lacquered faces.

5.4.2.2 Transfer a 100-ml aliquot of the solution to the 500-ml wide neck conical flask, add 75 ml of concentrated hydrochloric acid and 10 ml of ferric chloride solution and dilute to the 200 ml mark with water. Continue the reduction and titration as in **5.4.1.2** and **5.4.1.3** using the standard potassium iodate-iodide solution 2 as titrant.

5.4.2.3 Remove the lacquer from the specimens used in **5.4.2.1** by swabbing with cotton wool soaked in acetone. Place six of the discs with the unstripped surface uppermost in a circle on the platinum wire and continue as above.

5.4.3 *For hot dipped tinplate*

5.4.3.1 Take the clean specimen (*see* **2.2**) from eight sheets sampled as in Table 7 of IS : 1993-1982. Place the spiral of platinum wire centrally in a shallow dish (*see* Fig. 2). Place six of the discs in a circle on the platinum wire and carefully pour 150 ml of dilute hydrochloric acid into the dish. As soon as the coating is completely dissolved from both the faces, leaving steel surfaces exposed, transfer the acid quantitatively to 1 000-ml volumetric flask. Wash twice with 25 ml of water, transferring the washings to the flask. Repeat this whole procedure with successive lots of discs combining the acid and washings in the same volumetric flask, finally diluting to the mark with water.

5.4.3.2 Transfer a 100 ml aliquot of the solution to a 500-ml wide neck conical flask add 55 ml of concentrated hydrochloric acid and 10 ml ferric chloride solution and dilute to the 200 ml mark with water. Continue the reduction and titration as in **5.4.1.2** and **5.4.1.3** using the standard potassium iodate solution 3 as titrant.

5.5 *Expression of Result*

5.5.1 The value of the average coating mass is obtained from the following formula:

$$\text{Mass of tin coating in g/m}^2 = \frac{T \times V \times 10^6}{A}$$

where

T = tin titre (grams of tin/ml) for potassium iodate-iodide solution used,

V = volume of potassium iodate-iodide solution-used for titration, and

A = total specimen area in mm^2.

EXPLANATORY NOTE

This standard was first published in 1959 and subsequently revised in 1966 and 1977. In this revision, the weight loss method and Bendix method have been incorporated for determining mass of tin coating on hot dip tin plate and electrolytic tinplate. Weight loss method included in this revision may be used for more than 5·6 g/m^2 by mass of tin coating and referee method may be used for less than 5·6 g/m^2 by mass of tin coating.

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